

YMC-Triart C18를 이용한 컬럼 스위칭 LC-MS/MS법에 의한 인간 혈청 중 PFAS 분석

U210519A

유기불소화합물인 폴리 및 퍼플루오로알킬화합물(PFAS)은 발수성, 내열성 등 다양한 특성을 가지고 있어 광범위한 분야에서 사용되고 있습니다. 그러나, 건강에의 악영향이 염려되어 최근 세계적으로 조사·연구를 해 규제도 도입되고 있습니다.

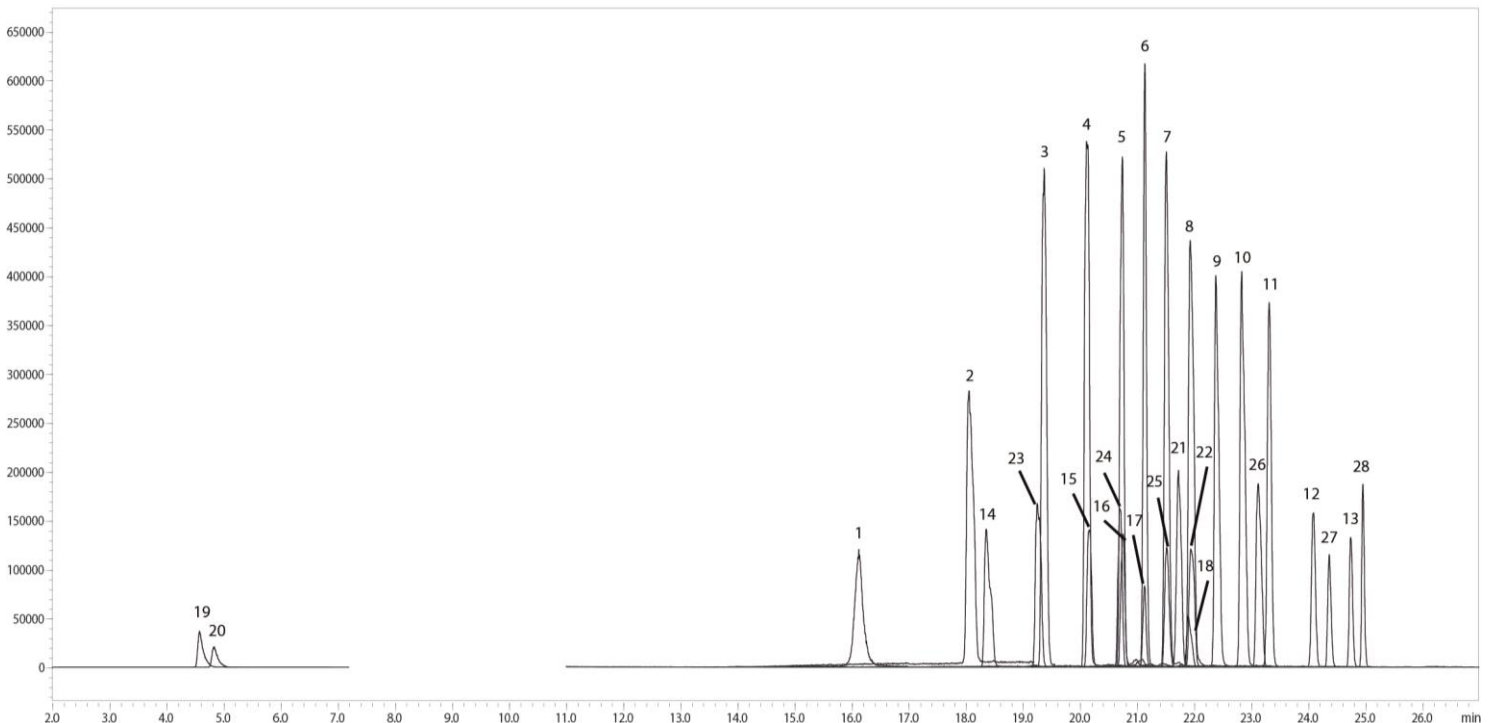
본 데이터 시트에서는 Nakayama 등에 의해 보고1)된 인간 혈청 중 28종류의 PFAS의 고감도 고수루트 분석 예를 소개합니다. 자동고상추출(SPE) 컬럼을 삽입한 컬럼 스위칭 LC-MS/MS를 사용한 메서드로, 분석 컬럼으로서 YMC-Triart C18이 사용되고 있습니다.

분석대상물

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|---|---|
| 1. Perfluorobutanoic acid (PFBA) | 17. Perfluorooctane sulphonic acid (PFOS) |
| 2. Perfluoropentanoic acid (PFPA) | 18. Perfluorodecane sulphonic acid (PFDS) |
| 3. Perfluorohexanoic acid (PFHxA) | 19. <i>N</i> -Methyl perfluorooctane sulphonamide (MeFOSA-M) |
| 4. Perfluoroheptanoic acid (PFHpA) | 20. <i>N</i> -Ethyl perfluorooctane sulphonamide (EtFOSA-M) |
| 5. Perfluorooctanoic acid (PFOA) | 21. <i>N</i> -Methyl perfluorooctane sulphonamido acetic acid (MeFOSA-A) |
| 6. Perfluorononanoic acid (PFNA) | 22. <i>N</i> -Ethyl perfluorooctane sulphonamido acetic acid (EtFOSA-A) |
| 7. Perfluorodecanoic acid (PFDA) | 23. 4:2 Fluorotelomer sulphonic acid (4:2 FTS) |
| 8. Perfluoroundecanoic acid (PFUnA) | 24. 6:2 Fluorotelomer sulphonic acid (6:2 FTS) |
| 9. Perfluorododecanoic acid (PFDoA) | 25. 8:2 Fluorotelomer sulphonic acid (8:2 FTS) |
| 10. Perfluorotridecanoic acid (PFTrDA) | 26. 6:2 Polyfluoroalkyl phosphoric acid diester (6:2 diPAP) |
| 11. Perfluorotetradecanoic acid (PFTeDA) | 27. 8:2 Polyfluoroalkyl phosphoric acid diester (8:2 diPAP) |
| 12. Perfluorohexadecanoic acid (PFHxDA) | 28. Perfluorooctane sulphonamido ethanol-based phosphate ester (diSAmPAP) |
| 13. Perfluorooctadecanoic acid (PFODA) | |
| 14. Perfluorobutane sulphonic acid (PFBS) | |
| 15. Perfluorohexane sulphonic acid (PFHxS) | |
| 16. Perfluoroheptane sulphonic acid (PFHpS) | |

크로마토그램

중성 부근의 pH이동상에서 중성화합물(피크 19, 20)을 분석한 후 알칼리성 이동상으로 전환하여 이온성 화합물을 분석하고 있습니다. Triart C18은 내구성이 우수하여 알칼리성 조건에서도 안정적인 분석이 가능합니다.



분석조건

Column : YMC-Triart C18 (3 μm, 12 nm)
 [Guard column] 10 X 2.1 mmI.D.
 [Analytical column] 100 X 2.1 mmI.D.

Temperature : 40°C

Detection : negative ESI

Injection : 400 μL

Eluent

Sample : standard solution (4 ng/mL)

LC Pump A : 2.5 mM ammonium acetate

Sample load

LC Pump B1 : methanol

LC pump D1 : water

LC Pump B2 : 2.5 mM ammonium acetate in
 95% methanol

On-line SPE column wash

LC Pump C : 0.1% ammonia in methanol

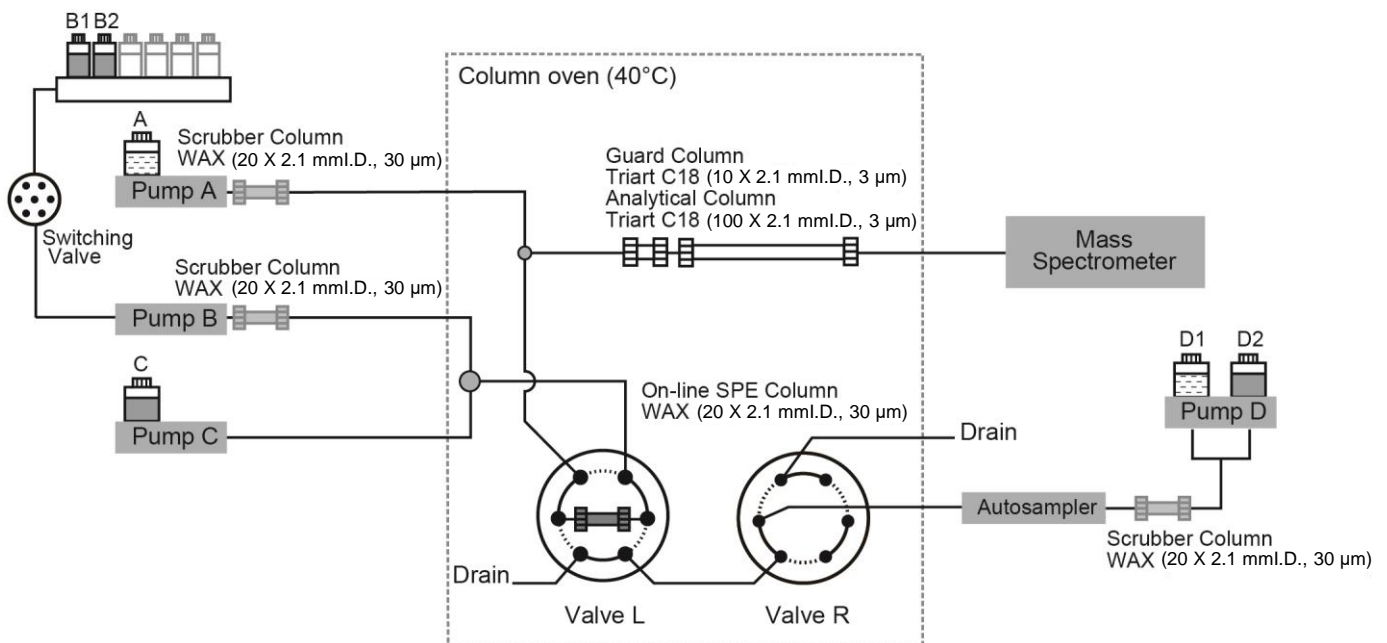
LC pump D2 : 0.5% ammonia in 90% methanol

System : LCMS-8060 (Shimadzu Corporation)

Time (min)	Ternary gradient				Flow rate Pump A+B+C (ml/min)	Binary gradient		Flow rate Pump D (ml/min)	Valve position	
	Pump A (%)	Pump B1 (%)	Pump B2 (%)	Pump C (%)		Pump D1 (%)	Pump D2 (%)		Valve L	Valve R
0.00	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	1	0
2.00	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	0	1
2.01	12.5	87.5	0.0	0.0	0.3	0.0	0.0	0.0	0	1
4.00	12.5	87.5	0.0	0.0	0.3	0.0	0.0	0.0	0	1
5.50	12.5	87.5	0.0	0.0	0.3	0.0	0.0	0.0	1	0
5.51	12.5	0.0	87.5	0.0	0.3	0.0	0.0	0.0	1	0
7.00	12.5	0.0	87.5	0.0	0.3	0.0	0.0	0.0	1	0
7.01	92.5	0.0	0.0	7.5	0.5	0.0	0.0	0.0	1	0
7.50	92.5	0.0	0.0	7.5	0.5	0.0	0.0	0.0	0	1
10.00	92.5	0.0	0.0	7.5	0.5	0.0	0.0	0.0	0	1
10.01	92.5	0.0	0.0	7.5	0.3	0.0	0.0	0.0	0	1
18.00	45	0.0	47.5	7.5	0.3	0.0	0.0	0.0	0	1
20.00	27.5	0.0	65	7.5	0.3	0.0	0.0	0.0	1	0
20.01	*a	0.0	*a	12.5	0.3	0.0	100	1.0	1	0
24.00	*a	0.0	*a	12.5	0.3	0.0	100	1.0	1	0
24.01	*a	0.0	*a	12.5	0.3	100	0.0	1.0	1	0
25.00	0.0	0.0	87.5	12.5	0.3	100	0.0	1.0	1	0
25.01	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	1	0
27.00	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	1	0

*a 20.00–25.00 min linear gradient (Pump A + Pump B2)

장치의 구성



Courtesy of Dr. Shoji F. Nakayama, National Institute for Environmental Studies

Reference 1)

Shoji F. Nakayama et al. Poly- and perfluoroalkyl substances in maternal serum: Method development and application in Pilot Study of the Japan Environment and Children's Study, Journal of Chromatography A 1618 (2020) 460933